

## FUNCTIONALIZED SILICAS

### Introduction and Background

The present invention relates to functionalized silicas, a process for their preparation and their use.

- 5 It is known to react silicon dioxide obtained by flame hydrolysis and with a surface area of 40 to 200 m<sup>2</sup>/g with 3-methacryloxypropyltrimethoxysilane. The resulting silicon dioxide is then coated with a further shell of (meth)acrylate polymers and subsequently employed in dental compositions (EP 0 142 784 A1).

### Summary of the Invention

- 10 The present invention provides functionalized silicas, characterized by functional groups fixed on the surface, the groups being 3-methacryloxypropylsilyl and/or glycidyloxypropylsilyl.

- The present invention also provides a process for the preparation of the functionalized silicas, which is characterized in that a silica is sprayed optionally first with water or dilute  
15 acid and then with a surface modification reagent or a mixture of several surface modification reagents in a suitable mixing vessel, with intensive mixing, the components are optionally re-mixed for 15 to 30 minutes and heat-treated at a temperature of 100 to 400 °C over a period of 1 to 6 h.

- A silica prepared pyrogenically by the route of flame hydrolysis of SiCl<sub>4</sub> can preferably be  
20 employed as the silica. Such pyrogenic silicas are known from Ullmanns Enzyklopädie der technischen Chemie [Ullmanns Encyclopaedia of Industrial Chemistry], 4th edition, volume 21, page 464 (1982).

In a preferred embodiment of the invention, a pyrogenic silica with a surface area of approx. 200 m<sup>2</sup>/g can be employed (Aerosil® 200).

- 25 Monomeric substances, such as 3-methacryloxypropyltrialkoxysilane and/or glycidyloxypropyltrialkoxysilane, wherein alkoxy can be methoxy, ethoxy and/or propoxy, can be employed as the surface modification reagent.

The amount of silane can be metered with respect to the silica such that no or only a small excess results. The excess silane can optionally be removed during the heat treatment.

The silica according to the invention can be employed in solvent-containing coatings, for example 2-component polyurethane coatings.

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#### **Detailed Description of Invention**

The functionalized silicas according to the invention have the following advantages:  
When used in solvent-containing coatings, such as, 2-component polyurethane coatings, the scratch resistance of the coating surface is increased.

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According to the invention, the pyrogenically prepared silicas according to table 1 can be employed as the silica for the silanization.

Table 1

## Physico-chemical data of AEROSIL®

Test method		AEROSIL 90	AEROSIL 130	AEROSIL 150	AEROSIL 200	AEROSIL 300	AEROSIL 380	AEROSIL OX 50	AEROSIL TT 600
Behaviour towards water	hydrophilic								
Appearance	loose white powder								
BET surface area <sup>1)</sup>	m <sup>2</sup> /g	90±5	130±25	150±15	200±25	300±30	380±30	50±15	200±50
Average primary particle size	nm	20	16	14	12	7	7	40	40
Tamped density approx. values <sup>2)</sup>	g/l	80	50	50	50	50	50	130	60
Compacted goods (added "V")	g/l	120	120	120	120	120	120		
VV goods (added "VV") <sup>12)</sup>	g/l			50/75	50/75	50/75			
Loss on drying <sup>3)</sup> (2 hours at 105 °C) on leaving supply works	%	<1.0	<1.5	<0.59	<1.5	<1.5	<2.0	<1.5	<2.5
Loss on ignition <sup>4)</sup> 7) (2 hours at 1000 °C)	%	<1	<1	<1	<1	<2	<2.5	<1	<2.5
pH <sup>5)</sup>		3.7-4.7	3.7-4.7	3.7-4.7	3.7-4.7	3.7-4.7	3.7-4.7	3.8-4.8	3.6-4.5
SiO <sub>2</sub> <sup>6)</sup>	%	>99.8	>99.8	>99.8	>99.8	>99.8	>99.8	>99.8	>99.8
Al <sub>2</sub> O <sub>3</sub> <sup>6)</sup>	%	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.08	<0.05
Fe <sub>2</sub> O <sub>3</sub> <sup>6)</sup>	%	<0.003	<0.003	<0.003	<0.003	<0.003	<0.003	<0.01	<0.003
TiO <sub>2</sub> <sup>6)</sup>	%	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03
HCl <sup>9)</sup>	%	<0.025	<0.025	<0.025	<0.025	<0.025	<0.025	<0.025	<0.025
Sieve residue <sup>8)</sup> (Mocker method, 45 µm)	%	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.2	<0.05
Drum size (net) <sup>11)</sup>	kg	10	10	10	10	10	10	10	10

- 1) in accordance with DIN 66131
- 2) in accordance with DIN ISO 787/XI, JIS K 5101/18 (not sieved)
- 3) in accordance with DIN ISO 787/II, ASTM D 280, JIS K 5101/21
- 4) in accordance with DIN 55921, ASTM D 1208, JIS K 5101/23
- 5) in accordance with DIN ISO 787/IX, ASTM D 1208, JIS K 5101/24
- 6) in accordance with DIN ISO 787/XVIII, JIS K 5101/20

- 7) based on the substance dried for 2 hours at 105 °C
- 8) based on the substance ignited for 2 hours at 1000 °C
- 9) special packaging protecting against moisture
- 10) HCl content is a constituent of the loss on ignition
- 11) V goods are supplied in sacks of 20 kg
- 12) VV goods are currently supplied only from the Rheinfelden works